CHAPTER 1

INTRODUCTION

1.1 Research Background

The past decade has witnessed tremendous effort and progress in the field of carbon nanotubes. Ever since the discovery of carbon nanotubes by Iijima (1991), it has captured the attention of researchers worldwide. Understanding their unique properties and exploring their potential applications have been a main driving force for this area.

Throughout history, the allotropes of carbon have played a number of important roles in technology. In ancient times, diamond was celebrated for its hardness and beauty, and carbon black was used as a colorant. The industrial age brought greater interest in graphite and related carbon materials as a source of carbon vapour in arc-lamps and as clean-burning fuels. Graphite-like carbon materials are now widely used for their unique mechanical, electrical and thermal properties.

Very small diameter (less than 10nm) carbon filaments were prepared in the 1970's and 1980's by the decomposition of hydrocarbons at high temperatures (Dresselhaus and Avouris, 2001). Direct stimulus to study carbon filaments of very small diameters more systematically came from the discovery of fullerenes by Kroto and Smalley.

In December 1990 at a carbon-carbon composites workshop, papers were given on the status of fullerene research by Smalley, the discovery of a new synthesis method for the efficient production of fullerenes and a review of carbon fibers research by M.S. Dresselhaus. Discussions at the workshop stimulated Smalley to speculate about the existence of carbon nanotubes of dimensions comparable to C_{60}. These conjectures were later followed up in August by an oral presentation at a fullerene workshop by Dresselhaus on the symmetry proposed for carbon nanotubes capped at either end by fullerene hemispheres (Saito *et al.*, 1998).

However, the real breakthrough on carbon nanotube research came with the experimental observation of carbon nanotubes in 1991 by Iijima of the NEC Laboratory in Tsukuba, Japan using High-Resolution Transmission Electron Microscopy (HRTEM) (Iijima, 1991). It was this work which bridged the gap between experimental observation and the theoretical framework of carbon nanotubes in relation to fullerenes and as theoretical examples of 1D system. Since the pioneering work by Iijima, the study of carbon nanotubes has progressed rapidly.

1.2 Problem Statement

The main hindrance to employing carbon nanotubes (CNTs) in real world is the inability to control the growth of the nanotubes and to grow bulk amounts of carbon nanotubes. There are three main techniques to grow carbon nanotubes: arc-discharge, laser ablation and chemical vapour deposition (CVD). The first two methods are high temperature processes that produce high quality CNTs, but they cannot grow mass quantities of nanotubes within a reasonable amount of time. The CVD technique is able to grow bulk amounts of nanotubes and arrays of multi-walled carbon nanotubes (MWNTs). However, these nanotubes contain a vast amount of defects along the length of the tubes due to the relatively low synthesis temperature of 600 – 1200 °C.

Nevertheless, some progress has been recently obtained, the chemical vapour deposition (CVD) has been modified by applying various supported metals catalysts in the production of CNTs. The catalytic chemical vapour deposition (CVD) method supplies CNTs in high yield and low costs, but also at controlling the CNTs characteristics and morphologies. Being a catalytic process, the combinations of transition metals and support can be changed depending on the characteristics required, such as the alignment and diameter of the nanotubes. The CCVD synthesis of CNTs can be carried out at low temperature and ambient pressure.

1.3 Research Objectives

This research is intended to synthesize carbon nanotubes (CNTs) of high yield and purity at economical cost. Therefore, this research is conducted to achieve the following primary objectives:

1. To produce carbon nanotubes (CNTs) using Catalytic Chemical Vapour Deposition (CCVD) method.

2. To study the effects of supported catalysts in the synthesis of carbon nanotubes (CNTs).

3. To characterise the supported catalysts and carbon nanotube yields chemically and physically.

4. To identify the best catalyst-support combination to catalyze the carbon nanotubes growth.

1.4 Scopes of Study

In order to achieve the objectives, this research is focusing on the following scopes:

(i) Designing and fabricating an effective Catalytic Chemical Vapour Deposition (CCVD) system contains a fixed bed flow reactor which facilitates the production of CNTs.

(ii) Preparing supported catalysts by using alumina (Al_2O_3) beads, molecular sieves (MS) beads and anodic aluminium oxide (AAO) template as catalyst supports and cobalt (Co) or ferrum (Fe) as metal catalysts with impregnation or dip coat techniques. Applying calcination treatment to enhance the activity of the catalysts.

(iii) Producing CNTs through catalyze pyrolysis of acetylene (C_2H_2) at optimum temperature, reaction time and gas flow rate.

(iv) Characterising the supported catalysts and CNT yields using X-ray Diffraction (XRD) technique to reveal the active catalyst sites and effects of calcination.

(v) Investigating the morphologies and topologies of the supported catalysts and the CNT yields through Scanning Electron Microscopy (SEM), Field-Emission Scanning Electron Microscopy (FE-SEM) and Transmission Electron Microscopy (TEM) techniques.

(vi) Determining the surface composition of a sample (metal catalyst and carbon content) using Energy Dispersive X-Ray Analysis (EDAX) technique.

(vii) Comparing the performance of supported catalysts in the production of CNTs to figure out the best combination of support, catalyst, loading and treatment for the supported catalysts.